

PC 078003



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 13, 2005

Subject: Sulfuryl Fluoride. Evaluation of the Proposed Fumigation of Food Processing Establishments. Summary of Analytical Chemistry and Residue Data. Petition Number 3F6573.

DP Number: 317730

Decision Number: 305993

PC Code: 078003

MRID Numbers: 45899201

40 CFR 180. 575 & 145

Chemical Class: Fumigant

From: Michael Doherty, Ph.D., Chemist
Registration Action Branch II
Health Effects Division (7509C)

A handwritten signature in black ink, appearing to read "Michael Doherty".

Through: Richard A. Loranger, Ph.D., Branch Senior Scientist
Registration Action Branch II
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A handwritten signature in black ink, appearing to read "R. Loranger".

To: Dan Kenny, Meredith Laws
Insecticide-Rodenticide Branch
Registration Division (7505C)

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Executive Summary

Sulfuryl fluoride is being proposed as a methyl bromide replacement to control pests in food processing establishments. Sulfuryl fluoride is a fumigant and, in the form of ProFume™, is formulated as 99+% active ingredient. The fumigation rate for sulfuryl fluoride is the product of the fumigant concentration and exposure time. Double fumigations are recommended for insect infestations where eggs may be present, with the second fumigation timed to control newly hatched, immature stages. The maximum cumulative target rate is 1500 mg·hr/L for normal atmospheric fumigations and 200 mg·hr/L for vacuum fumigations. The proposed label specifies that all food commodities be actively aerated for a minimum of 24 hours prior to the foods entering commerce. The petitioner, Dow Agrosciences, has requested tolerances as follows:

Commodity	Requested Tolerance, ppm	
	Sulfuryl Fluoride	Fluoride Ion
Flavorings, leavening agents (except yeast), dry garlic, dry onion, dry pepper, baking powder, baking soda	0.05	8
Other herbs, spices, chili pepper	0.3	70
Salt, sugars, high-fructose corn syrup	0.02	2
Peanuts	0.2	13
Coffee, cocoa beans	0.8	12
Dried legume vegetables (beans, peas, soybean, etc.)	0.02	6
Powdered milk, powdered cheese	1.5	3
All other processed foods	1.2	70

Tolerances are currently established for residues of sulfuryl fluoride in/on cereal grains, tree nuts, and dried fruits (40 CFR 180.575); and for residues of inorganic fluoride resulting from the use of either sulfuryl fluoride or cryolite (40 CFR 180.145). Sulfuryl fluoride is highly reactive and breaks down to form sulfate and fluoride anions. Parent sulfuryl fluoride and the fluoride anion are the residues of concern for the tolerance expression and for risk assessment purposes.

To support the requested uses, Dow Agrosciences has submitted residue data for sulfuryl fluoride and fluoride anion from a number of finished food products (chips, cookies, etc.) as well as foods considered to be "key" ingredients (salt, sugar, powdered milk, etc.). Foods were fumigated at approximately the maximum label rate (1500 mg·hr/L) and allowed to aerate for 24 hours prior to residue analysis. Fumigation, aeration, and storage were all done at 30°C in order to maximize the potential conversion of sulfuryl fluoride to fluoride anion, as demonstrated by previously submitted data (MRID 45170401). For finished foods, items were fumigated in an open configuration (i.e., a box or other open container) as well as in their original packaging. Key ingredients were fumigated only in the open configuration.

Separate analytical methods for each residue of concern are available for most

2

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

commodities; however, the data submitted to support this petition shows that the methods are not suitable for all commodities that may be treated. Furthermore, storage stability data for fluoride were not submitted and there is concern that fluoride may have reacted with food components during storage and become "bound." There is evidence from previous storage stability studies with fluoride (MRID 45510302) that this may occur.

Residues of sulfuryl fluoride were highly dependent on the nature of the fumigated material and ranged from <0.004 ppm to approximately 2 ppm. Similarly, fluoride residues were dependent on the commodity and ranged from <1 to approximately 820 ppm. Generally, commodities with higher protein and/or fat content have higher residues of sulfuryl fluoride or fluoride (an extreme case being powdered eggs). For a number of finished products, the residues of sulfuryl fluoride in the packaged configuration were greater than in the open configuration. In all such cases, the packaging contained a polymer film, either as a bag liner or as lined paper. The phenomena was not mirrored in the fluoride residue levels. HED does not have a satisfactory theory to explain these observations at this time.

Regulatory Recommendations and Residue Chemistry Deficiencies

Method performance leaves a high degree of uncertainty surrounding residues of sulfuryl fluoride in Oreo[®] cookies, powdered eggs, and baking soda; and for residues of fluoride in white cake mix, pet foods, parsley, and baking powder. Given the transient nature of sulfuryl fluoride residues and the potential for fluoride to serve as a marker compound, HED does not believe that the lack of a universal method for sulfuryl fluoride warrants development of a new sulfuryl fluoride method. HED is, however, concerned about the lack of performance of the fluoride method for some commodities and the fluoride storage stability issue noted above. HED notes that the use of a total fluoride analysis method would resolve both the method and the storage stability issues and recommends that the petitioner investigate and, if necessary, validate a total fluoride method, using representative commodities from all crop groups and animal commodities (meat, fat, milk, eggs). Further, HED recommends that the petitioner consult with the HED prior to the onset of any such investigation. HED is recommending that the registration, if granted, be made conditional on the proposal of a more universal fluoride method, acceptance of that method by the Agency, and submission of residue data collected from control and fumigated representative commodities. As of 6/2/05, the Analytical Chemistry Laboratory had not received reference standards of either sulfuryl fluoride or a suitable fluoride salt. Due to the nature of sulfuryl fluoride, the laboratory is not requesting that a standard be submitted at this time; however, a reference standard for fluoride should be provided.

Based on the data available at this time, that the following tolerances be established:

Commodity	Tolerance, ppm	
	Sulfuryl Fluoride	Fluoride Ion
All processed food commodities not otherwise listed	2.0	70
Cattle, meat, dried	0.01	40
Cheese	2.0	5.0

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

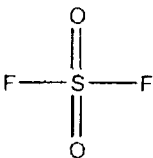
Cocoa bean, postharvest	0.2	20
Coconut, postharvest	1.0	40
Coffee, postharvest	1.0	15
Cottonseed, postharvest	0.5	70
Eggs, dried	1.0	900
Ginger, postharvest	0.5	70
Ham	0.02	20
Herbs and Spices, Group 19, postharvest	0.5	70
Milk, powdered	2.0	5.0
Nut, pine, postharvest	0.2	20
Peanut, postharvest	0.5	15
Rice, flour, postharvest	0.05	45
Vegetables, Legume, Group 6, postharvest	0.5	70

Background

Dow AgroSciences has requested permanent tolerances for residues of sulfuryl fluoride and fluoride anion resulting from the fumigation of food storage, processing, and handling establishments. Sulfuryl fluoride is a potential methyl bromide replacement.

Sulfuryl fluoride is a highly volatile compound with a boiling point of -55°C and a vapor pressure of 11552 Torr (20°C). At 20°C , sulfuryl fluoride has a vapor density of 4.3 g/L (heavier than air) and is both colorless and odorless. The log K_{ow} is estimated to be 0.41. Sulfuryl fluoride has a very low solubility in water (0.075 g/100 g). Solubility in other solvents are 0.78 g/100 g in Wesson oil, 1.74 g/100 g in acetone, and 2.12 g/100 g in chloroform.

Table 1. Test Compound Nomenclature.

Chemical Structure		F ⁻
Common name	Sulfuryl fluoride	Fluoride
Company experimental name	Sulfuryl fluoride	Fluoride
IUPAC name	Sulfuryl Fluoride	Fluoride
CAS name	Sulfuryl Fluoride	Fluoride
CAS #	2699-79-8	16984-48-8
Molecular Formula	F ₂ O ₂ S	F ⁻
Molecular weight	102.056	19.0

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

End-use product/EP	Profume; Vikane (gas fumigant)
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Table 2. Physicochemical Properties of the Technical Grade Test Compound. Properties for fluoride were not available		
Parameter	Value	Reference
Melting point/range	-136°C	Vikane MSDS
Boiling point	-55°C	Vikane MSDS
pH	Not Provided	
Density (20°C)	4.3 g/L	Vikane MSDS
Water solubility	1.67 g/L	Vikane MSDS
Solvent solubility	Vegetable oil: 0.78 g/100g Acetone: 1.74 g/100 g Chloroform: 2.2 g/100 g	Vikane Chemical Fact Sheet
Vapor pressure (20°C)	11552 mm Hg (Torr)	Vikane MSDS
Dissociation constant (pK _a)	Not Provided	
Octanol-water partition coefficient Log(K _{ow})	Not Provided	
UV/visible absorption spectrum	Not Provided	

860.1200 Directions for Use

Table 3. Summary of Directions for the Post-harvest Use of Sulfuryl Fluoride from the Proposed Label.						
Applic. Timing, Type, and Equip.	Formulation [EPA Reg. No.]	Applic. Rate (mg hr/L)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (mg hr/L)	Aeration (hours)	Use Directions and Limitations
Fumigation of sealed mills, warehouses, chambers, and other storage, processing, or food handling structures	ProFume [62719-XXX]	1500 (ambient pressure)	2	1500 (ambient pressure)	24	Food commodities must be aerated for 24 hours prior to entering commerce.
		200 (vacuum fumigation)		200 (vacuum fumigation)		

The proposed label has sufficient information to allow the Agency to evaluate the residue trials in light of the proposed use patterns.

860.1300 Nature of the Residue - Plants

DER: None

MARC Decision Memo: None

Nature of the residue studies have not been submitted for sulfuryl fluoride. Following application, sulfuryl fluoride breaks down to form sulfate and fluoride anion. Sulfate is not of toxicological concern. Fluoride anion is of toxicological concern due to potential skeletal fluorosis. The residues of concern for both tolerance enforcement and risk assessment purposes are sulfuryl fluoride and fluoride anion. The lack of nature of the residue studies is not

5

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

considered to be a deficiency and no further data are required to fulfill this guideline.

860.1300 Nature of the Residue - Livestock

DER: None

MARC Decision Memo: None

As with plants, sulfuryl fluoride would be expected to hydrolyze to form sulfate and fluoride anions. The residues of concern in livestock are sulfuryl fluoride and the fluoride anion; however, due to the nature of the sulfuryl fluoride molecule, HED believes it is unlikely that secondary residues of sulfuryl fluoride will occur in livestock commodities. The lack of nature of the residue studies is not considered to be a deficiency and no further data are required to fulfill this guideline.

860.1340 Residue Analytical Methods

DER: M. Doherty, 1/13/04, MRID 45632902 (Sulfuryl Fluoride; 45632902.der.wpd)
M. Doherty, 1/13/04, MRID 45603901 (Fluoride anion; 45603901.der.wpd)

Analytical methods for both sulfuryl fluoride and fluoride anion are discussed in the previous residue chemistry summary document (D309015, M. Doherty, 10/12/04). Although both HED and the Analytical Chemistry Branch (Biological and Economic Analysis Division) have some reservations about the methods and how they were validated, they were deemed acceptable by the Agency. The method for sulfuryl fluoride has a limit of quantitation (LOQ) of 0.004 ppm. The method for fluoride has a LOQ of 1 ppm.

Concurrent recovery data submitted with the subject matter study volume raise questions about the suitability of the sulfuryl fluoride method for Orco[®] cookies (23% recovery), powdered eggs (11% recovery), and baking soda (10% recovery). For Oreo[®] cookies and powdered eggs, residues of fluoride were high following fumigation. A rapid conversion of sulfuryl fluoride to fluoride in the presence of these commodities, as suggested by the higher fluoride levels, would account for the low recovery of the sulfuryl fluoride. Since residues of fluoride were not measured in the sulfuryl fluoride fortified concurrent recovery samples, the suitability of the sulfuryl fluoride method for Oreo[®] cookies and powdered eggs remains in question. The registrant surmises that the low recovery of sulfuryl fluoride from baking soda is due to the baking soda itself raising the pH in the solution that results during extraction, and that this higher pH raises the solubility of sulfuryl fluoride resulting in less material being in the headspace of the extraction vessel. Regardless of the mechanism, the method does not appear to be adequate for baking soda.

Concurrent recovery of fluoride was highly variable, particularly at the lower fortification level (5 ppm). Recovery of fluoride was quite variable at both tested fortification levels for powdered non-fat milk (40-114%), parsley (39-79%), and baking soda (40-164%). Average recovery of fluoride falls into the acceptable range (70-120%) for most commodities. Exceptions to this are Doritos[®] (177%), white cake mix (59%), all four pet foods (17-67%), parsley (59%), and baking powder (not quantifiable). Based on control samples of Doritos[®], the high recovery in that food is not due to a high background concentration of fluoride anion. There is no apparent

6

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

explanation for the low recoveries noted in the other six commodities.

Conclusions. The methods for sulfuryl fluoride and fluoride appear to be acceptable for some of commodities included in the study design and unacceptable for others. Given the wide scope of foods that may be treated under a food handling establishment use, HED does not believe that the analytical methods are universally adequate. HED believes that the use of a total fluoride analysis method would resolve the method issue and recommends that the petitioner investigate and, if necessary, validate a total fluoride method, using representative commodities from all crop groups and animal commodities (meat, fat, milk, eggs). The currently proposed method is satisfactory in the interim.

860.1360 Multiresidue Methods

DER: None

Multiresidue method studies have not been submitted to the Agency. Based on their physicochemical properties, neither sulfuryl fluoride nor fluoride anion are likely to be suitable for the multiresidue techniques currently in use. The lack of multiresidue studies is not considered to be a deficiency and no further data are required to fulfill this guideline.

860.1380 Storage Stability

DER: None

The petitioner has not submitted storage stability data to support the residue data submitted as MRID 45899201. Samples were analyzed for residues of sulfuryl fluoride as soon as possible following the aeration interval. Samples used for fluoride analysis were stored, at 30°C, for up to 19 days.

Conclusions. Storage stability data to support the sulfuryl fluoride residue data are not required. HED is concerned, however, with the lack of storage stability data for fluoride. Fluoride anion is a highly reactive molecule that may react with food components and become "bound." Data from the wheat storage stability study (MRID 45510302) show a significant "loss" of fluoride with time in wheat flour. Fluoride appeared to be stable in other commodities in that study. The adoption of a total fluoride method, as recommended by HED (see above) would obviate the need for fluoride storage stability data.

860.1400 Water, Fish, and Irrigated Crops

DER: None

There are no proposed uses for sulfuryl fluoride that would result in residues in fish or irrigated crops. A discussion of the 860.1400 guideline is not germane to this petition.

860.1460 Food Handling

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

DER: M. Doherty, 6/2/05, MRID 45899201

Dow AgroSciences LLC has submitted residue data from 2003 for sulfuryl fluoride and fluoride on finished products and "key" ingredients. Finished products included Cheezits®, Fritos®, Doritos®, spaghetti, egg noodles, chocolate cake mix, white cake mix, corn flakes, granola, flour tortilla, corn tortilla, Pecan Sandies®, peanut butter cookies, coconut flakes, Oreo® cookies, Alpo® dog food, Purina Puppy Chow®, 9 Lives® cat food, Whiska's® cat food, ham, cocoa beans, coffee beans, ground coffee, and beef jerky. Items that were classified as key ingredients were powdered nonfat milk, powdered whole milk, powdered cheese, powdered eggs, garlic powder, onion powder, peppercorns, parsley, baking powder, baking soda, salt, sugar, basil, in-shell peanuts, and dried chili peppers. Most finished products were fumigated in their retail packaging (packaged configuration) and removed from their packaging (open configuration). Cocoa beans, coffee, and ham, as well as all of the key ingredients, were fumigated in the open configuration only. The data reflect residues of sulfuryl fluoride and fluoride in/on these foods that result from a single fumigation at a rate of 1414 to 1734 g hr/m³ at 30°C followed by a 23- to 27-hour aeration period at 5 to 10 L/min.

The analytical method for sulfuryl fluoride is a GC/ECD headspace method. Method validation and concurrent recoveries were deemed to be acceptable for this study except for Oreo® cookies, powdered eggs and baking soda. The method for fluoride uses a fluoride-specific ion electrode with a double-known addition quantitation technique. Fluoride method validation and concurrent recoveries were deemed to be acceptable for this study except for Doritos®, white cake mix, Alpo® dog food, Purina Puppy Chow®, 9 Lives® cat food, Whiska's® cat food, parsley and baking powder. The limits of quantitation (LOQ) for the methods are 4 µg/kg for sulfuryl fluoride and 1 µg/g for fluoride. The limits of detection (LOD) of the methods was not reported.

Samples for sulfuryl fluoride analysis were taken as soon as possible after the aeration period and immediately extracted and analyzed. Samples for fluoride analysis were stored at 30°C for 9-19 days. Based on our knowledge of fluoride anion behavior and limited data from wheat, HED is concerned about potential formation of "bound" fluoride residues during the storage interval.

Residues of sulfuryl fluoride were highly dependent on the nature of the fumigated material and ranged from <0.004 ppm to approximately 2 ppm. Similarly, fluoride residues were dependent on the commodity and ranged from <1 to approximately 820 ppm. Generally, commodities with higher protein and/or fat content have higher residues of sulfuryl fluoride or fluoride (an extreme case being powdered eggs). For a number of finished products, the residues of sulfuryl fluoride in the packaged configuration were greater than in the open configuration. In all such cases, the packaging contained a polymer film, either as a bag liner or as lined paper. The phenomena was not mirrored in the fluoride residue levels. HED does not have a satisfactory theory to explain these observations at this time.

Residue levels of sulfuryl fluoride and fluoride anion are summarized in Tables 2 and 3, below. Residue data have been corrected based on concurrent recovery values.

9

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Table 4. Summary of Sulfuryl Fluoride Residue Data from Foods Fumigated with Sulfuryl Fluoride.

Commodity	Configuration	n	Sulfuryl Fluoride Residue Level, ppm			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Cheezits®	Open	2	< 0.004	< 0.004	< 0.004	0.000
Cheezits®	Packaged	2	0.021	0.029	0.025	0.005
Fritos®	Open	2	< 0.004	0.004	0.003	0.002
Fritos®	Packaged	2	< 0.004	0.005	0.004	0.002
Doritos®	Open	2	0.123	0.153	0.138	0.021
Doritos®	Packaged	2	0.009	0.406	0.208	0.281
Spaghetti	Open	2	< 0.004	< 0.004	< 0.004	0.000
Spaghetti	Packaged	2	< 0.004	< 0.004	< 0.004	0.000
Egg Noodles	Open	2	< 0.004	< 0.004	< 0.004	0.000
Egg Noodles	Packaged	2	< 0.004	< 0.004	< 0.004	0.000
Chocolate Cake Mix	Open	2	< 0.004	< 0.004	< 0.004	0.000
Chocolate Cake Mix	Packaged	2	< 0.004	0.013	0.007	0.007
White Cake Mix	Open	2	0.013	0.020	0.017	0.005
White Cake Mix	Packaged	2	0.026	0.038	0.032	0.009
Corn Flakes	Open	2	0.277	1.993	1.135	1.213
Corn Flakes	Packaged	2	0.085	0.087	0.086	0.002
Granola	Open	2	0.011	0.032	0.022	0.015
Granola	Packaged	2	0.106	0.136	0.121	0.021
Flour Tortilla	Open	2	0.004	0.004	0.004	0.000
Flour Tortilla	Packaged	2	0.011	0.025	0.018	0.010
Corn Tortilla	Open	2	0.019	0.047	0.033	0.020
Corn Tortilla	Packaged	2	0.005	0.006	0.005	0.001
Pecan Sandies®	Open	2	0.061	0.065	0.063	0.003
Pecan Sandies®	Packaged	2	0.101	0.199	0.150	0.069
Peanut Butter Cookies	Open	2	< 0.004	< 0.004	< 0.004	0.000
Peanut Butter Cookies	Packaged	2	< 0.004	0.011	0.006	0.006
Coconut Flakes	Open	2	0.808	0.991	0.900	0.129
Coconut Flakes	Packaged	2	0.166	0.203	0.185	0.026
Oreo® Cookies	Open	2	0.161	0.197	0.179	0.025
Oreo® Cookies	Packaged	2	0.075	0.129	0.102	0.038
Alpo® Dog Food	Open	2	0.049	0.101	0.075	0.037
Alpo® Dog Food	Packaged	1	1.208	1.208	1.208	
Purina Puppy Chow®	Open	2	0.250	0.318	0.284	0.048
Purina Puppy Chow®	Packaged	1	1.864	1.864	1.864	
9 Lives® Cat Food	Open	2	0.023	0.026	0.024	0.002
9 Lives® Cat Food	Packaged	1	0.027	0.027	0.027	

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Table 4. Summary of Sulfuryl Fluoride Residue Data from Foods Fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Sulfuryl Fluoride Residue Level, ppm			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Whiska's® Cat Food	Open	2	0.100	0.109	0.105	0.006
Whiska's® Cat Food	Packaged	1	1.175	1.175	1.175	--
Coffee Beans	Open	2	0.011	0.011	0.011	0.000
Ground Coffee	Open	2	0.799	0.832	0.816	0.023
Beef Jerky	Open	2	< 0.004	0.007	0.005	0.004
Beef Jerky	Packaged	2	< 0.004	< 0.004	< 0.004	0.000
Powdered Nonfat Milk2	Open	2	< 0.004	< 0.004	< 0.004	0.000
Powdered Whole Milk	Open	2	1.439	1.466	1.453	0.019
Powdered Cheese	Open	2	0.344	0.472	0.408	0.091
Powdered Eggs	Open	2	0.253	0.634	0.444	0.269
Garlic Powder	Open	2	< 0.004	< 0.004	< 0.004	0.000
Onion Powder	Open	2	< 0.004	< 0.004	< 0.004	0.000
Peppercorns	Open	2	0.012	0.018	0.015	0.004
Parsley	Open	2	0.120	0.204	0.162	0.059
Baking Powder	Open	2	0.027	0.036	0.032	0.007
Baking Soda	Open	2	< 0.004	< 0.004	< 0.004	0.000
Salt	Open	2	< 0.004	< 0.004	< 0.004	0.000
Sugar	Open	2	< 0.004	< 0.004	< 0.004	0.000
Basil	Open	2	0.008	0.013	0.010	0.004
Peanuts	Open	2	0.082	0.209	0.145	0.090
Chilis	Open	2	0.229	0.264	0.247	0.025
Ham	Open	2	< 0.004	< 0.004	< 0.004	0.000
Cocoa Beans	Open	2	0.113	0.121	0.117	0.006

^aThe Highest Average Field Trial (HAFT) for each table entry is equal to the maximum residue value due to the experimental design.

^bThe median (50th percentile) is not provided since there were, at most, only two residue values reported.

Shaded entries in the table are for commodity-analyte combinations with unacceptable method performance based on concurrent recovery.

Table 5. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Cheezits®	Open	2	1.00	1.00	1.00	0.00
Cheezits®	Packaged	2	1.00	1.00	1.00	0.00
Fritos®	Open	2	4.42	5.61	5.02	0.84
Fritos®	Packaged	2	1.23	1.23	1.23	0.00
Doritos®	Open	2	5.94	5.95	5.95	0.01

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Table 5. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Doritos®	Packaged	2	2.28	2.37	2.33	0.06
Spaghetti	Open	2	< 1	< 1	< 1	0.00
Spaghetti	Packaged	2	< 1	< 1	< 1	0.00
Egg Noodles	Open	2	17.50	17.80	17.65	0.21
Egg Noodles	Packaged	2	< 1	< 1	< 1	0.00
Chocolate Cake Mix	Open	2	6.72	7.86	7.29	0.81
Chocolate Cake Mix	Packaged	2	1.49	2.63	2.06	0.81
White Cake Mix	Open	2	2.77	7.28	5.03	3.19
White Cake Mix	Packaged	2	2.77	3.94	3.36	0.83
Corn Flakes	Open	2	1.38	5.27	3.33	2.75
Corn Flakes	Packaged	2	< 1	< 1	< 1	0.00
Granola	Open	2	16.01	18.71	17.36	1.91
Granola	Packaged	2	1.01	2.37	1.69	0.96
Flour Tortilla	Open	2	25.80	35.10	30.45	6.58
Flour Tortilla	Packaged	2	9.71	10.10	9.91	0.28
Corn Tortilla	Open	2	26.25	31.25	28.75	3.54
Corn Tortilla	Packaged	2	11.30	14.95	13.13	2.58
Pecan Sandies®	Open	2	15.70	17.10	16.40	0.99
Pecan Sandies®	Packaged	2	1.18	1.66	1.42	0.34
Peanut Butter Cookies	Open	2	56.73	67.73	62.23	7.78
Peanut Butter Cookies	Packaged	2	1.23	42.93	22.08	29.49
Coconut Flakes	Open	2	34.88	35.48	35.18	0.42
Coconut Flakes	Packaged	2	8.37	9.30	8.84	0.66
Oreo® Cookies	Open	2	25.60	27.00	26.30	0.99
Oreo® Cookies	Packaged	2	< 1	< 1	< 1	0.00
Alpo® Dog Food	Open	2	3.08	3.10	3.09	0.01
Alpo® Dog Food	Packaged	1	2.73	2.73	2.73	-
Purina Puppy Chow®	Open	2	12.40	16.00	14.20	2.55
Purina Puppy Chow®	Packaged	1	19.20	19.20	19.20	-
9 Lives® Cat Food	Open	2	8.87	9.93	9.40	0.75
9 Lives® Cat Food	Packaged	1	8.32	8.32	8.32	-
Whiskas® Cat Food	Open	2	6.71	7.78	7.25	0.76
Whiskas® Cat Food	Packaged	1	4.93	4.93	4.93	-
Coffee Beans	Open	2	6.81	7.48	7.15	0.47
Ground Coffee	Open	2	10.00	11.58	10.79	1.12
Beef Jerky	Open	2	24.72	37.92	31.32	9.33

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Table 5. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Beef Jerky	Packaged	2	1.12	1.12	1.12	0.00
Powdered Nonfat Milk ²	Open	2	2.36	2.36	2.36	0.00
Powdered Whole Milk	Open	2	1.07	2.31	1.69	0.88
Powdered Cheese	Open	2	4.05	4.90	4.48	0.60
Powdered Eggs	Open	2	690.84	819.84	755.34	91.22
Garlic Powder	Open	2	15.80	15.80	15.80	0.00
Onion Powder	Open	2	1.17	1.17	1.17	0.00
Peppercorns	Open	2	5.36	6.55	5.96	0.84
Parsley	Open	2	48.15	56.25	52.20	5.73
Baking Powder	Open	0	-	-	-	-
Baking Soda	Open	2	14.10	15.60	14.85	1.06
Salt	Open	2	2.04	2.30	2.17	0.18
Sugar	Open	2	< 1	1.03	0.77	0.37
Basil	Open	2	48.83	53.53	51.18	3.32
Peanuts	Open	2	10.30	12.30	11.30	1.41
Chilis	Open	2	24.95	27.75	26.35	1.98
Ham	Open	2	17.43	18.73	18.08	0.92
Cocoa Beans	Open	2	12.30	14.01	13.16	1.21

^a The Highest Average Field Trial (HAFT) for each table entry is equal to the maximum residue value due to the experimental design.

^b The median (50th percentile) is not provided since there were, at most, only two residue values reported.

Shaded entries in the table are for commodity/analyte combinations with unacceptable method performance based on concurrent recovery.

Conclusions. Residue levels of sulfuryl fluoride and fluoride that result in foods following fumigation with sulfuryl fluoride are highly dependent on the properties of those foods, primarily fat and protein content. Furthermore, the performance of the analytical methods used in this study for sulfuryl fluoride and fluoride is not consistent across all of the foods tested and may be a factor in the observed large variability in the residue data. Fluoride may have reacted with food components during the interval that samples were stored, making it unavailable for analysis; thus uncorrected data may significantly under predict fluoride residues resulting from fumigation with sulfuryl fluoride. Data are inconclusive regarding the ability of polymer film packaging materials to serve as a barrier to sulfuryl fluoride gas.

860.1480 Meat, Milk, Poultry, and Eggs

DER: None

Studies describing residues of sulfuryl fluoride and fluoride anion in meat, milk, poultry, and eggs have not been submitted to the Agency. Due to the nature of the sulfuryl fluoride

Sulfuryl Fluoride Summary of Analytical Chemistry and Residue Data Barcode: D317730

molecule, HED believes it is unlikely that secondary residues of sulfuryl fluoride will occur in livestock commodities. The ability of fluoride anion to be transferred from animal feeds to livestock commodities is unclear. Following review of the previous petition for use of sulfuryl fluoride in grain milling establishments, HED requested that livestock feeding studies be done for the fluoride anion.

860.1500 Crop Field Trials

DER: None. Data submitted under 860.1500 for the current petition are presented above under Section 860.1460 Food Handling.

Conclusions. See Section 860.1460 above.

860.1520 Processed Food and Feed

DER: None

Conclusions. Data for processed food and feed are not pertinent to the requested use.

860.1650 Submittal of Analytical Reference Standards

As of 6/2/05, the Analytical Chemistry Laboratory had not received reference standards of either sulfuryl fluoride or a suitable fluoride salt. Due to the nature of sulfuryl fluoride, the laboratory is not requesting that a standard be submitted at this time; however, a reference standard for fluoride should be provided (pers. comm. C. Stafford, 6/2/05).

860.1850 Confined Accumulation in Rotational Crops

DER: None.

Conclusions. Data for rotational crops are not pertinent to the requested use.

860.1900 Field Accumulation in Rotational Crops

DER: None.

Conclusions. Data for rotational crops are not pertinent to the requested use.

860.1550 Proposed Tolerances

Tolerances are currently established for residues of sulfuryl fluoride in/on cereal grains, tree nuts, and dried fruits (40 CFR 180.575); and for residues of inorganic fluoride resulting from the use of either sulfuryl fluoride or cryolite (40 CFR 180.145). Sulfuryl fluoride is highly reactive and breaks down to form sulfate and fluoride anions. Parent sulfuryl fluoride and the fluoride anion are the residues of concern for both the tolerance expression and risk assessment purposes.

B

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Table 6. Tolerance Summary for Sulfuryl Fluoride			
Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Flavorings, leavening agents (except yeast), dry garlic, dry onion, dry pepper, baking powder, baking soda	0.05	None	Covered under "All processed food commodities not otherwise listed"
Other herbs, spices, chili pepper	0.3	0.5	Herbs and spices, group 19, postharvest
Salt, sugars, high-fructose corn syrup	0.02	None	Covered under "All processed food commodities not otherwise listed."
Peanuts	0.2	0.5	Peanut, postharvest
Coffee, cocoa beans	0.8	See below	Separate listings should be made for Coffee, postharvest and Cocoa bean, postharvest
Cocoa beans, postharvest	0.8	0.2	
Coffee, postharvest	0.8	1.0	
Dried legume vegetables (beans, peas, soybean, etc.)	0.02	0.5	Vegetables, legume, group 19, postharvest
Powdered milk, powdered cheese	1.5	See below	Separate listings should be made for milk, powdered and cheese
Cheese	1.5	2.0	
Milk, powdered	1.5	2.0	
All other processed foods	1.2	2.0	All processed food commodities not otherwise listed
Cattle, meat, dried	0.01	0.01	
Coconut, postharvest	1.0	1.0	
Egg	0.7	1.0	Eggs, dried
Ginger, postharvest	0.2	0.5	
Ham	0.01	0.02	
Nut, pine, postharvest	3.0	0.2	
Rice, flour, postharvest	0.08	0.05	
Grain, cereal forage, fodder, and straw, group 16, postharvest	2.0	None	No data to support tolerance
Grass, forage, fodder, and hay, group 17, postharvest	2.0	None	No data to support tolerance

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Animal Feed	2.0	None	Covered under "All processed food commodities not otherwise listed."

Table 7. Tolerance Summary for Fluoride			
Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Flavorings, leavening agents (except yeast), dry garlic, dry onion, dry pepper, baking powder, baking soda	8	None	Covered under "All processed food commodities not otherwise listed."
Other herbs, spices, chili pepper	70	70	Herbs and spices, group 19, postharvest
Salt, sugars, high-fructose corn syrup	2	None	Covered under "All processed food commodities not otherwise listed."
Peanuts	13	15	Peanut, postharvest
Coffee, cocoa beans	12	See below	Separate listings should be made for Coffee, postharvest and Cocoa bean, postharvest
Cocoa beans, postharvest	12	20	
Coffee, postharvest	12	15	
Dried legume vegetables (beans, peas, soybean, etc.)	6	70	Vegetables, legume, group 19, postharvest
Powdered milk, powdered cheese	3	See below	Separate listings should be made for milk, powdered and cheese
Cheese	3	5.0	
Milk, powdered	3	5.0	
All other processed foods	70	70	All processed food commodities not otherwise listed
Cattle, meat, dried	40	40	
Coconut, postharvest	40	40	
Egg	850	900	Eggs, dried
Ginger, postharvest	13	70	
Ham	20	20	

K

Sulfuryl Fluoride

Summary of Analytical Chemistry and Residue Data

Barcode: D317730

Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Nut, pine, postharvest	10	20	
Rice, flour, postharvest	98	45	
Grain, cereal forage, fodder, and straw, group 16, postharvest	130	None	No data to support tolerance
Grass, forage, fodder, and hay, group 17, postharvest	130	None	No data to support tolerance
Animal Feed	130	None	Covered under "All processed food commodities not otherwise listed."

Template Version November 2003



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Primary Evaluator

Date: 6/2/05

Michael Doherty, Ph.D., Chemist
Registration Action Branch 2, HED (7509C)

Approved by

Date: 6/2/05

Richard A. Loranger, Ph.D., Branch Senior Scientist
Registration Action Branch 2, HED (7509C)

This Data Evaluation Record was originally prepared by R. Vogrig of Health Canada's Pest Management Regulatory Agency and provided to EPA as a courtesy. It has been revised to reflect EPA/OPP/HED conclusions and current policies.

Note that this study was submitted and reviewed under the OPPTS 860.1500 guideline for a crop field trial even though the petitioned request is for a food handling establishment use.

STUDY REPORTS:

MRID 45899201. Rick, D. L., Krieger, S. M. and Mielke, M. S. (2003). Magnitude of the Terminal Fluoride Ion Level in Finished Food Products Fumigated with Sulfuryl Fluoride. Study Project Number: 021080. Unpublished study prepared by Dow AgroSciences LLC. 81 pages.

EXECUTIVE SUMMARY:

Dow AgroSciences LLC has submitted residue data from 2003 for sulfuryl fluoride and fluoride on finished products and "key" ingredients. Finished products included Cheezits[®], Fritos[®], Doritos[®], spaghetti, egg noodles, chocolate cake mix, white cake mix, corn flakes, granola, flour tortilla, corn tortilla, Pecan Sandies[®], peanut butter cookies, coconut flakes, Oreo[®] cookies, Alpo[®] dog food, Purina Puppy Chow[®], 9 Lives[®] cat food, Whiska's[®] cat food, ham, cocoa beans, coffee beans, ground coffee, and beef jerky. Items that were classified as key ingredients were powdered nonfat milk, powdered whole milk, powdered cheese, powdered eggs, garlic powder, onion powder, peppercorns, parsley, baking powder, baking soda, salt, sugar, basil, in-shell peanuts, and dried chili peppers. Most finished products were fumigated in their retail packaging (packaged configuration) and removed from their packaging (open configuration). Cocoa beans, coffee, and ham, as well as all of the key ingredients, were fumigated in the open configuration only. The data reflect residues of sulfuryl fluoride and fluoride in/on these foods that result from a single fumigation at a rate of 1414 to 1734 g hr/m³ at 30°C followed by a 23- to 27-hour aeration period at 5 to 10 L/min.

The analytical method for sulfuryl fluoride is a GC/ECD headspace method. Method validation and concurrent recoveries were deemed to be acceptable for this study except for Oreo[®] cookies, powdered eggs and baking soda. The method for fluoride uses a fluoride-specific ion electrode with a double-known addition quantitation technique. Fluoride method validation and concurrent recoveries were deemed to be acceptable for this study except for Doritos[®], white

17



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

cake mix, Alpo® dog food, Purina Puppy Chow®, 9 Lives® cat food, Whiska's® cat food, parsley and baking powder. The limits of quantitation (LOQ) for the methods are 4 µg/kg for sulfuryl fluoride and 1 µg/g for fluoride. The limits of detection (LOD) of the methods were not reported.

Samples for sulfuryl fluoride analysis were taken as soon as possible after the aeration period and immediately extracted and analyzed. Samples for fluoride analysis were stored at 30°C for 9-19 days. Based on our knowledge of fluoride anion behavior and limited data from wheat, HED is concerned about potential formation of "bound" fluoride residues during the storage interval.

Residues of sulfuryl fluoride were highly dependent on the nature of the fumigated material and ranged from <0.004 ppm to approximately 2 ppm. Similarly, fluoride residues were dependent on the commodity and ranged from <1 to approximately 820 ppm. Generally, commodities with higher protein and/or fat content have higher residues of sulfuryl fluoride or fluoride (an extreme case being powdered eggs). For a number of finished products, the residues of sulfuryl fluoride in the packaged configuration were greater than in the open configuration. In all such cases, the packaging contained a polymer film, either as a bag liner or as lined paper. The phenomena was not mirrored in the fluoride residue levels. HED does not have a satisfactory theory to explain these observations at this time.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the fumigation trial residue data are classified as scientifically acceptable for most of the commodities. Method performance leaves a high degree of uncertainty surrounding residues of sulfuryl fluoride in Oreo® cookies, powdered eggs, and baking soda; and for residues of fluoride in white cake mix, pet foods, parsley, and baking powder. Also, the lack of storage stability for fluoride is of concern, even with the short (<20 days) storage interval for these samples, due to the ability of the anion to bind with components of the samples, potentially reducing its extractability. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document (DP Barcode D312659).

COMPLIANCE:

Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided.

A. BACKGROUND INFORMATION

Sulfuryl fluoride is a gas fumigant pesticide being proposed as a methyl bromide replacement. The fumigation rate for sulfuryl fluoride is the product of the fumigant concentration and exposure time. For the current petition, the maximum target rate is 1500 mg-hr/L. The target rate may be achieved by manipulating both the in-air concentration of the

18



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

gas as well as the duration of the fumigation.

Table A.1. Test Compound Nomenclature.		
Chemical Structure	$\begin{array}{c} \text{O} \\ \parallel \\ \text{F}-\text{S}-\text{F} \\ \parallel \\ \text{O} \end{array}$	F
Common name	Sulfuryl fluoride	Fluoride
Company experimental name	Sulfuryl fluoride	Fluoride
IUPAC name	Sulfuryl Fluoride	Fluoride
CAS name	Sulfuryl Fluoride	Fluoride
CAS #	2699-79-8	16984-48-8
Molecular Formula	F ₂ O ₂ S	F
Molecular weight	102.056	19.0
End-use product/EP	Profume, Vikane (gas fumigant)	

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound. Properties for fluoride were not available.		
Parameter	Value	Reference
Melting point/range	-136°C	Vikane MSDS
Boiling point	-55°C	Vikane MSDS
pH	Not Provided	
Density (20°C)	4.3 g/L	Vikane MSDS
Water solubility	1.67 g/L	Vikane MSDS
Solvent solubility	Not Provided	
Vapor pressure (20°C)	11552 mm Hg (Torr)	Vikane MSDS
Dissociation constant (pK _a)	Not Provided	
Octanol/water partition coefficient Log(K _{ow})	Not Provided	
UV/visible absorption spectrum	Not Provided	

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

Table B.1.1. Trial Site Conditions.						
Trial Identification (City, State/Year)	Soil characteristics				Meteorological data	
	Type	%OM	pH	CEC meq/g	Overall daily/monthly rainfall range	Overall T°C range
Not applicable to post-harvest use.						

19



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

The in-laboratory study was performed in specially designed exposure chambers.

Table B.1.2. Study Use Pattern.

EP	Temperature (°C)	Exposure		Aeration	
		Time (hr)	Total Rate (mg hr/L)	Time (hr)	Total Rate (L/min)
ProFume	30±2.5	24	1414-1734	23-27	5-10

EP = End-use Product

Several commodities (2 to 6) were fumigated together in the fumigation chamber, but kept in separate containers. Key ingredients (powdered milk, sugar, salt, etc.) were fumigated under non-packaged conditions only. Finished foods (Doritos®, egg noodles, Purina Puppy Chow®, etc.) were fumigated under both packaged and unpackaged conditions. Non-packaged commodities were contained in open paper or a cardboard container. The packaged commodities were unopened as purchased from a retail grocery store. The retail packaging could be a polymer film, cardboard box or etc. Two fumigation and a control fumigation chambers were used. Each commodity was fumigated only once, with the replicate fumigations being conducted in two different chambers. Each chamber was made from a modified 25-gallon steel drum with an internal fan that was used during both the fumigation and aeration phases of the study. The control samples were handled in the same manner as the test samples. After completion of the exposure period, commodities were aerated with building air at 30°C. To maintain a constant level of sulfuryl fluoride and to compensate for the absorption of sulfuryl fluoride during the fumigation process, the chamber was dosed at 110 to 120% of the target 1500 mg·hr/L rate. The volume of sulfuryl fluoride used was determined by subtracting the volume occupied by the commodity from the volume of the chamber. The temperature in the control chamber during one fumigation test had risen to 39°C, but went back to 30°C after 1 hour. It was necessary to re-dose to maintain target exposure concentration in both chambers for the following commodities: Alpo® dog food, Purina Puppy Chow®, 9 Lives® cat food, Whiska's® cat food. Sulfuryl fluoride in one of the chambers decreased significantly during one exposure experiment and was not redone. To compensate for the loss of sulfuryl fluoride, the time of exposure was extended from 24 to 27 hours. All experiments were done at atmospheric pressure and at 30°C for both the fumigation and aeration phases.

Table B.1.3. Trial Numbers and Geographical Locations.

NAFTA Growing Region	Crop 1			Crop 2			Crop 3		
	Submitted	Requested		Submitted	Requested		Submitted	Requested	
		Canada	US		Canada	US		Canada	US
Not applicable to post-harvest use.									

B.2. Sample Handling and Preparation

20



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Following each fumigation and associated 24-hour aeration period, the chambers were opened and commodities sampled as quickly as possible for the determination of sulfuryl fluoride concentration. Fumigated commodities were then placed in a temperature-controlled incubator, maintained at 30°C, until the analysis of sulfuryl fluoride and fluoride residues were completed. Control commodities were also stored at 30°C in an incubator that was not used in any of the exposure experiments.

B.3. Analytical Methodology

To analyze sulfuryl fluoride residues, a duplicate 25-g sample (10-g for parsley) was homogenized in water in an airtight container. An aliquot of the gaseous head space was removed and analyzed for sulfuryl fluoride by gas chromatography (GC) using a ⁶³Ni electron capture detector. The detector response to sulfuryl fluoride was log curve linearized in the range of 30 to 150 mg sulfuryl/L with a coefficient of determination (r^2) 0.9988. This method has a limit of quantitation (LOQ) of 0.004 µg/g (ppm). The limit of detection (LOD) was not reported.

To analyze fluoride residues, a 10-g sample was macerated in a blender containing water and total ionic strength adjusting buffer. The extract was centrifuged and the fluoride residue in the aqueous solution was analyzed using a pH/fluoride ion meter equipped with a double-known addition calibration/measurement algorithm and a reference electrode. Analyses were done in duplicate. This method has a LOQ of 1 ppm. The LOD was not reported.

C. RESULTS AND DISCUSSION

The fumigations in this study were conducted at elevated temperatures (30°C) in an attempt to maximize the conversion of sulfuryl fluoride to fluoride anion in the fumigated commodities. Therefore, these data have the potential to under predict residues of sulfuryl fluoride following fumigation at lower temperatures. Based on the Agency's review of previous studies (MRID 45170401), this potential underestimation is not expected to be significant. The petitioner notes that the test commodities were chosen to represent a range of finished products and key ingredients that might be present in food processing facility or warehouses and focused on commodities that would likely be fumigated.

The analytical method for the analysis of sulfuryl fluoride gives concurrent recoveries that are rather variable, especially at the lower fortification level (0.01 ppm; Table C.1.1.). Variability in recovery is particularly high for parsley, basil, and ham. On average, recoveries are acceptable for all commodities except Oreo® cookies (23%), powdered eggs (11%), and baking soda (10%). For Oreo® cookies and powdered eggs, residues of fluoride were high following fumigation (Table C.3.). A rapid conversion of sulfuryl fluoride to fluoride in the presence of these commodities, as suggested by the higher fluoride levels, would account for the low recovery of the sulfuryl fluoride. Without proof of this, however, the suitability of the sulfuryl fluoride method for Oreo® cookies and powdered eggs remains in question. The registrant surmises that the low recovery of sulfuryl fluoride from baking soda is due to the baking soda

21



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

itself raising the pH in the solution that results during extraction, and that this higher pH raises the solubility of sulfuryl fluoride resulting in less material being in the headspace of the extraction vessel. Regardless of the mechanism, the method does not appear to be adequate for baking soda.

As with sulfuryl fluoride, variability in the recovery of fluoride is often significantly higher at the lower (5 ppm) fortification level. We note that recovery of fluoride from powdered non-fat milk, parsley, and baking soda are quite variable at both tested fortification levels (Table C.1.2.). Recovery of fluoride falls into the acceptable range (70-120%) for most commodities. Exceptions to this are Doritos® (177%), white cake mix (59%), all four pet foods (17-67%), parsley (59%), and baking powder (not quantifiable). Based on control samples of Doritos®, the high recovery in that food is not due to a high background concentration of fluoride anion. There is no apparent explanation for the low recoveries noted in the other six commodities. For baking powder, a relationship between fortification level and electrode response could not be established; therefore, quantitation of residues in baking powder was not possible.

Reported residues values for sulfuryl fluoride and fluoride anion were corrected based on average concurrent recovery values for each commodity, including those listed above as being problematic.

Residues of sulfuryl fluoride were analyzed almost immediately following the aeration interval; therefore, storage stability data are not required to support the residue data. Fluoride analysis was completed within 19 days of fumigation (Table C.2.). HED notes that for the duration of aeration and storage, samples were maintained at 30°C in order to maximize the conversion of sulfuryl fluoride to fluoride anion. HED is concerned that during the storage interval, especially at the elevated temperature, fluoride could combine with food components and not be "available" for analysis by specific-ion electrode. Data from wheat (MRID 45510302) suggest that the potential "loss" of fluoride in storage may be significant.

Residues of sulfuryl fluoride and fluoride in finished foods and key ingredients vary widely depending on the nature of the matrix (Table C.3.). Foods with a higher fat content tend to have higher residues of sulfuryl fluoride. Foods with a higher protein content tend to have higher fluoride residues (powdered eggs being an extreme example). Data regarding the effectiveness of packaging as a barrier to sulfuryl fluoride are equivocal. In some instances residues of sulfuryl fluoride are lower in packaged foods in comparison to the same food fumigated in the open configuration. However, higher residues of sulfuryl fluoride in packaged foods versus open configuration is not uncommon in the dataset. In all such cases, the packaging contained a polymer film, either as a bag liner or as lined paper. The relative level of sulfuryl fluoride in open versus packaged foods is not mirrored in the fluoride residue data, which we would expect to be the case if the higher residues in packaged foods was due to compromised integrity of the packaging material. HED does not have a satisfactory theory to explain these observations at this time.

Table C.1.1. Summary of Concurrent Recoveries of Sulfuryl Fluoride from Finished Foods.
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Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Matrix	Spike level (µg/kg)	Sample size (n)	Mean Recoveries (%)	Average Mean Recovery (%)
Cheezits [®]	10	4	92.9, 79.7	86.3
	80	4	86.3, 91.6	89.0
Fritos [®]	10	4	95.5, 79.5	87.5
	80	4	81.4, 92.0	86.7
Doritos [®]	10	4	84.7, 101.0	92.9
	80	4	95.9, 101.0	98.5
Spaghetti	10	4	96.4, 79.3	87.9
	80	4	95.4, 96.6	96.0
Egg Noodles	10	4	111.0, 120.0	115.5
	80	4	117.0, 80.3	98.7
Chocolate Cake Mix	10	4	90.3, 95.5	92.9
	80	4	97.4, 96.6	97.0
White Cake Mix	10	4	118.0, 98.5	108.3
	80	4	87.9, 94.5	91.2
Corn Flakes	10	4	78.4, 114.0	96.2
	80	4	97.8, 98.1	98.0
Granola	10	4	109.0, 84.8	96.9
	80	4	96.8, 88.9	92.9
Flour Tortilla	10	4	83.4, 88.5	86.0
	80	4	119.0, 101.0	110.0
Corn Tortilla	10	4	91.1, 93.8	92.5
	80	4	101.0, 101.0	101.0
Pecan Sandies [®]	10	4	88.6, 65.9	77.3
	80	4	76.5, 72.4	74.5
Peanut Butter Cookies	10	4	80.6, 129.0	104.8
	80	4	95.1, 92.9	94.0
Coconut Flakes	10	4	119.0, 153.0	136.0
	80	4	101.0, 89.3	95.2
Oreo [®] Cookies	10	4	<LOQ, <LOQ	
	80	4	23.8, 22.4	23.1
Alpo [®] Dog Food	10	4	107.0, 90.4	98.7
	80	4	96.6, 97.9	97.3
Purina Puppy Chow [®]	10	4	65.3, 97.0	81.2
	80	4	100.0, 110.0	105.0
9 Lives [®] Cat Food	10	4	106.0, 99.3	102.7
	80	4	104.0, 95.1	99.6
Whiska's [®] Cat Food	10	4	78.1, 103.0	90.6
	80	4	94.6, 93.0	93.8
Coffee Beans	10	4	95.1, 112.0	103.6

83



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

	80	4	97.4, 93.3	95.4
Ground Coffee	10	4	89.2, 101.0	95.1
	80	4	96.8, 94.3	95.6
Beef Jerky	10	4	105.0, 95.5	100.3
	80	4	84.3, 101.0	92.7
Powdered Nonfat Milk	10	4	113.0, 96.6	104.8
	80	4	91.6, 97.3	94.5
Powdered Whole Milk	10	4	116.0, 94.3	105.2
	80	4	93.1, 109.0	101.1
Powdered Cheese	10	4	92.6, 108.0	100.3
	80	4	87.8, 99.0	93.4
Powdered Eggs	10	4	<LOQ, <LOQ	-
	80	4	13.5, 9.13	11.3
Garlic Powder	10	4	101.0, 95.0	98.0
	80	4	96.1, 103.0	99.6
Onion Powder	10	4	96.0, 95.5	95.8
	80	4	96.9, 114.0	105.5
Peppercorns	10	4	106.0, 91.3	98.7
	80	4	88.9, 91.9	90.4
Parsley	10	4	45.8, 66.9	56.4
	80	4	91.0, 64.9	78.0
Baking Powder	10	4	90.9, 91.4	91.2
	80	4	94.9, 93.5	94.2
Baking Soda	10	4	<LOQ, <LOQ	-
	80	4	9.18, 11.7	10.4
Salt	10	4	93.7, 82.6	88.2
	80	4	98.3, 91.9	95.1
Sugar	20	2	96.5	96.5
	80	4	108.0, 80.0	94.0
Basil	10	4	111.0, 81.8	96.4
	80	4	87.4, 68.5	78.0
Peanuts	10	4	95.2, 99.4	97.3
	80	4	102.0, 99.9	101.0
Chilis	10	4	107.0, 94.2	100.6
	80	4	87.1, 98.6	92.9
Ham	10	4	103.0, 87.9	95.5
	80	4	104.0, 84.5	94.3
Cocoa Beans	10	4	86.7, 88.7	87.7
	80	4	88.0, 82.6	85.3

Individual recoveries are based on an average of two replicates.

24



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.1.2. Summary of Concurrent Recoveries of Fluoride from Finished Foods.

Matrix	Spike level (µg/g)	Sample size (n)	Mean Recoveries (%)	Average Mean Recovery (%)
Cheezits®	0	1	NA	-
	5	2	114.0, 113.0	113.5
	50	2	90.6, 92.8	91.7
Fritos®	0	1	NA	-
	5	2	109.0, 117.0	113.0
	50	2	90.6, 96.2	93.4
Doritos®	0	1	NA	-
	5	2	178.0, 176.0	177.0
	50	2	108.0, 146.0	127.0
Spaghetti	0	1	NA	-
	5	2	109.0, 122.0	115.5
	50	2	94.8, 106.0	100.4
Egg Noodles	0	1	NA	-
	5	2	119.0, 115.0	117.0
	50	2	91.4, 99.4	95.4
Chocolate Cake Mix	0	1	NA	-
	5	2	79.6, 77.2	78.4
	50	2	75.0, 76.6	75.8
White Cake Mix	0	1	NA	-
	5	2	53.6, 46.8	50.2
	50	2	59.6, 58.0	58.8
Corn Flakes	0	1	NA	-
	5	2	120.0, 123.0	121.5
	50	2	92.4, 100.0	96.2
Granola	0	1	NA	-
	5	2	109.0, 118.0	113.5
	50	2	94.0, 106.0	100.0
Flour Tortilla	0	1	NA	-
	5	2	62.2, 44.4	53.3
	50	2	85.0, 86.0	85.5
Corn Tortilla	0	1	NA	-
	5	2	104.0, 96.8	100.4
	50	2	82.0, 98.2	90.1
Pecan Sandies®	0	1	NA	-
	5	2	118.0, 129.0	123.5
	50	2	85.6, 110.0	97.8
Peanut Butter Cookies	0	1	NA	-
	5	2	99.4, 71.4	85.4

25



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.1.2. Summary of Concurrent Recoveries of Fluoride from Finished Foods.

Matrix	Spike level (µg/g)	Sample size (n)	Mean Recoveries (%)	Average Mean Recovery (%)
	50	2	95.2, 101.0	98.1
Coconut Flakes	0	1	NA	-
	5	2	106.0, 133.0	119.5
	50	2	98.6, 70.6	84.6
Oreo [®] Cookies	0	1	NA	-
	5	2	119.0, 118.0	118.5
	50	2	89.0, 104.0	96.5
Alpo [®] Dog Food	0	1	NA	-
	5	2	41.4, 53.8	47.6
	50	2	43.2, 58.0	50.6
Purina Puppy Chow [®]	0	1	NA	-
	5	2	<LOQ, <LOQ	-
	50	2	49.6, 80.6	65.1
9 Lives [®] Cat Food	0	1	NA	-
	5	2	17.2, 17.0	17.1
	50	2	42.8, 39.0	40.9
Whiska's [®] Cat Food	0	1	NA	-
	5	2	62.4, 59.6	61.0
	50	2	65.2, 69.2	67.2
Coffee Beans	0	1	NA	-
	5	2	133.0, 118.0	125.5
	50	2	89.8, 92.4	91.1
Ground Coffee	0	1	NA	-
	5	2	119.0, 120.0	119.5
	50	2	91.6, 97.6	94.6
Beef Jerky	0	1	NA	-
	5	2	158.0, 118.0	138.0
	50	2	103.0, 103.0	103.0
Powdered Nonfat Milk	0	1	NA	-
	5	2	246.0, 39.8	142.9
	50	2	104.0, 96.0	100.0
Powdered Whole Milk	0	1	NA	-
	5	2	114.0, 114.0	114.0
	50	2	99.4, 91.2	95.3
Powdered Cheese	0	1	NA	-
	5	2	114.0, 162.0	138.0
	50	2	99.0, 85.0	92.0
Powdered Eggs	0	1	NA	-

2/10



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD ILA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.1.2. Summary of Concurrent Recoveries of Fluoride from Finished Foods.				
Matrix	Spike level (µg/g)	Sample size (n)	Mean Recoveries (%)	Average Mean Recovery (%)
	5	2	108.0, 92.2	100.1
	50	2	68.0, 89.6	78.8
	800	4	97.3, 95.8, 101.0, 116.0	96.6
Garlic Powder	0	1	NA	-
	5	2	158.0, 198.0	178.0
	50	2	107.0, 90.0	98.5
Onion Powder	0	1	NA	-
	5	2	113.0, 118.0	115.5
	50	2	89.2, 91.6	90.4
Peppercorns	0	1	NA	-
	5	2	112.0, 114.0	113.0
	50	2	91.4, 93.8	92.6
Parsley	0	1	NA	-
	5	2	39.2, 78.8	59.0
	50	2	43.2, 60.8	52.0
Baking Powder	0	1	NA	-
	5	2	Note	-
	50	2	-	-
Baking Soda	0	1	NA	-
	5	2	164.0, 66.0	115.0
	50	2	40.2, 96.4	68.3
Salt	0	1	NA	-
	5	2	97.8, 100.0	98.9
	50	2	91.0, 94.8	92.9
Sugar	0	1	NA	-
	5	2	104.0, 109.0	106.5
	50	2	94.8, 95.4	95.1
Basil	0	1	NA	-
	5	2	138.0, 182.0	160.0
	50	2	109.0, 110.0	109.5
Peanuts	0	1	NA	-
	5	2	121.0, 127.0	124.0
	50	2	92.8, 103.0	97.9
Chilis	0	1	NA	-
	5	2	104.0, 105.0	104.5
	50	2	92.0, 96.6	94.3
Ham	0	1	NA	-
	5	2	68.0, 76.4	72.2

27



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.1.2. Summary of Concurrent Recoveries of Fluoride from Finished Foods.

Matrix	Spike level (µg/g)	Sample size (n)	Mean Recoveries (%)	Average Mean Recovery (%)
	50	2	96.8, 110.0	103.4
Cocoa Beans	0	1	NA	-
	5	2	137.0, 218.0	177.5
	50	2	108.0, 96.4	102.2

NA - Not Applicable.

Note - A relationship between fluoride fortification level and electrode response was not found; therefore quantitation of residues is not possible.

Individual mean recoveries were based in a mean of two replicates.

Table C.2. Summary of Storage Conditions.

Matrix (RAC or Extract)	Storage Temp. (°C)	Actual Storage Duration (Days)	Interval of Demonstrated Storage Stability
Sulfuryl Fluoride			
Finished Foods	30	Same day	None
Fluoride			
Finished Foods	30	19	Concurrent

Table C.3. Residue Data from Finished Foods fumigated with Sulfuryl Fluoride.

Commodity (Control Fluoride Level)	Configuration	Average Rate	Sulfuryl Fluoride, ppm	Fluoride, ppm ^a
Cheezits® (1 ppm)	Open	1729.5	<0.004, <0.004	1, 1
	Packaged	1730	0.0212, 0.0288	1, 1
Fritos® (1.23 ppm)	Open	1729.5	0.00446, <0.004	4.42, 5.61
	Packaged	1730	<0.004, 0.00545	1.23, 1.23
Doritos® (<1 ppm)	Open	1729.5	0.123, 0.153	5.94, 5.95
	Packaged	1730	0.406, 0.00928	2.37, 2.28
Spaghetti (<1 ppm)	Open	1687	<0.004, <0.004	<1, <1
	Packaged	1687.5	<0.004, <0.004	<1, <1
Egg Noodles (<1 ppm)	Open	1687	<0.004, <0.004	17.5, 17.8
	Packaged	1687.5	<0.004, <0.004	<1, <1
Chocolate Cake Mix (1.49 ppm)	Open	1687	<0.004, <0.004	6.72, 7.86
	Packaged	1687.5	<0.004, 0.0125	1.49, 2.63
White Cake Mix (2.77 ppm)	Open	1687	0.0204, 0.013	2.77, 7.28
	Packaged	1687.5	0.0381, 0.0255	3.94, 2.77
Corn Flakes (<1 ppm)	Open	1604	1.993, 0.277	5.27, 1.38
	Packaged	1604.5	0.0874, 0.0848	<1, <1
Granola (1.01 ppm)	Open	1604	0.011, 0.0323	18.71, 16.01
	Packaged	1604.5	0.106, 0.136	2.37, 1.01

98



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.3. Residue Data from Finished Foods fumigated with Sulfuryl Fluoride.				
Commodity (Control Fluoride Level)	Configuration	Average Rate	Sulfuryl Fluoride, ppm	Fluoride, ppm*
Flour Tortilla (1.4 ppm)	Open	1604	0.00423, 0.00437	35.1, 25.8
	Packaged	1604	0.0106, 0.0254	10.1, 9.71
Corn Tortilla (1.95 ppm)	Open	1604	0.0472, 0.0194	26.25, 31.25
	Packaged	1604	0.00584, 0.00495	11.3, 14.95
Pecan Sandies® (<1 ppm)	Open	1601.5	0.0652, 0.0614	17.1, 15.7
	Packaged	1601.5	0.101, 0.199	1.18, 1.66
Peanut Butter Cookies (1.23 ppm)	Open	1601.5	<0.004, <0.004	67.73, 56.73
	Packaged	1601.5	0.0108, <0.004	42.93, 1.23
Coconut Flakes (1.28 ppm)	Open	1601.5	0.991, 0.808	34.88, 35.48
	Packaged	1601.5	0.166, 0.203	8.37, 9.3
Oreo® Cookies (<1 ppm)	Open	1601.5	0.161, 0.197	27, 25.6
	Packaged	1601.5	0.0749, 0.129	<1, <1
Alpo® Dog Food (<1 ppm)	Open	1558	0.049, 0.101	3.1, 3.08
	Packaged	1578	1.208	2.73
Purina Puppy Chow® (12.4 ppm)	Open	1558	0.25, 0.318	12.4, 16
	Packaged	1578	1.864	19.2
9 Lives® Cat Food (3.88 ppm)	Open	1558	0.0256, 0.0226	8.87, 9.93
	Packaged	1578	0.027	8.32
Whiskas® Cat Food (3.17 ppm)	Open	1558	0.1, 0.109	7.78, 6.71
	Packaged	1578	1.175	4.93
Coffee Beans (1.25 ppm)	Open	1634	0.0106, 0.0109	6.81, 7.48
Ground Coffee (1.18 ppm)	Open	1634	0.832, 0.799	11.58, 10
Beef Jerky (1.12 ppm)	Open	1634	0.00739, <0.004	37.92, 24.72
	Packaged	1634	<0.004, <0.004	1.12, 1.12
Powdered Nonfat Milk (2.36 ppm)	Open	1430	<0.004, <0.004	2.36, 2.36
Powdered Whole Milk (1.07 ppm)	Open	1430	1.466, 1.439	1.07, 2.31
Powdered Cheese (1.75 ppm)	Open	1430	0.344, 0.472	4.05, 4.9
Powdered Eggs (1.84 ppm)	Open	1430	0.253, 0.634	819.84, 690.84
Garlic Powder (8.31 ppm)	Open	1430	<0.004, <0.004	15.8, 15.8
Onion Powder	Open	1430	<0.004, <0.004	1.17, 1.17

29



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.3. Residue Data from Finished Foods fumigated with Sulfuryl Fluoride.				
Commodity (Control Fluoride Level)	Configuration	Average Rate	Sulfuryl Fluoride, ppm	Fluoride, ppm*
(1.17 ppm)				
Peppercorns (1.08 ppm)	Open	1488.5	0.0182, 0.012	5.36, 6.55
Parsley (8.45 ppm)	Open	1488.5	0.204, 0.12	56.25, 48.15
Baking Powder (Not Determined)	Open	1488.5	0.0267, 0.0364	---
Baking Soda (11.7 ppm)	Open	1488.5	<0.004, <0.004	14.1, 15.6
Salt (<1 ppm)	Open	1488.5	<0.004, <0.004	2.04, 2.3
Sugar (<1 ppm)	Open	1488.5	<0.004, <0.004	1.03, <1
Basil (4.93 ppm)	Open	1582.5	0.00798, 0.013	48.83, 53.53
Peanuts (<1 ppm)	Open	1582.5	0.0819, 0.209	10.3, 12.3
Chilis (1.45 ppm)	Open	1582.5	0.229, 0.264	27.75, 24.95
Ham (3.23 ppm)	Open	1689.5	<0.004, <0.004	17.43, 18.73
Cocoa Beans (2.31 ppm)	Open	1689.5	0.121, 0.113	14.01, 12.3

*Fluoride levels were originally reported as corrected for background fluoride. Fluoride levels in this table include background levels (i.e., reported residues are background plus any additional residue as a result of treatment). Residue levels are corrected for concurrent recovery values.

Shaded entries in the table are for commodity/analyte combinations with unacceptable method performance based on concurrent recovery.

Table C.4.1. Summary of Sulfuryl Fluoride Residue Data from Foods Fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Sulfuryl Fluoride Residue Level, ppm*			
			Min, ppm	Max, ppm ^b	Mean, ppm ^b	SD, ppm
Cheezits®	Open	2	< 0.004	< 0.004	< 0.004	0.000
Cheezits®	Packaged	2	0.021	0.029	0.025	0.005
Fritos®	Open	2	< 0.004	0.004	0.003	0.002
Fritos®	Packaged	2	< 0.004	0.005	0.004	0.002
Doritos®	Open	2	0.123	0.153	0.138	0.021
Doritos®	Packaged	2	0.009	0.406	0.208	0.281
Spaghetti	Open	2	< 0.004	< 0.004	< 0.004	0.000
Spaghetti	Packaged	2	< 0.004	< 0.004	< 0.004	0.000



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.4.1. Summary of Sulfuryl Fluoride Residue Data from Foods Fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Sulfuryl Fluoride Residue Level, ppm ^a			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Egg Noodles	Open	2	< 0.004	< 0.004	< 0.004	0.000
Egg Noodles	Packaged	2	< 0.004	< 0.004	< 0.004	0.000
Chocolate Cake Mix	Open	2	< 0.004	< 0.004	< 0.004	0.000
Chocolate Cake Mix	Packaged	2	< 0.004	0.013	0.007	0.007
White Cake Mix	Open	2	0.013	0.020	0.017	0.005
White Cake Mix	Packaged	2	0.026	0.038	0.032	0.009
Corn Flakes	Open	2	0.277	1.993	1.135	1.213
Corn Flakes	Packaged	2	0.085	0.087	0.086	0.002
Granola	Open	2	0.011	0.032	0.022	0.015
Granola	Packaged	2	0.106	0.136	0.121	0.021
Flour Tortilla	Open	2	0.004	0.004	0.004	0.000
Flour Tortilla	Packaged	2	0.011	0.025	0.018	0.010
Corn Tortilla	Open	2	0.019	0.047	0.033	0.020
Corn Tortilla	Packaged	2	0.005	0.006	0.005	0.001
Pecan Sandies [®]	Open	2	0.061	0.065	0.063	0.003
Pecan Sandies [®]	Packaged	2	0.101	0.199	0.150	0.069
Peanut Butter Cookies	Open	2	< 0.004	< 0.004	< 0.004	0.000
Peanut Butter Cookies	Packaged	2	< 0.004	0.011	0.006	0.006
Coconut Flakes	Open	2	0.808	0.991	0.900	0.129
Coconut Flakes	Packaged	2	0.166	0.203	0.185	0.026
Oreo [®] Cookies	Open	2	0.161	0.197	0.179	0.025
Oreo [®] Cookies	Packaged	2	0.075	0.129	0.102	0.038
Alpo [®] Dog Food	Open	2	0.049	0.101	0.075	0.037
Alpo [®] Dog Food	Packaged	1	1.208	1.208	1.208	
Purina Puppy Chow [®]	Open	2	0.250	0.318	0.284	0.048
Purina Puppy Chow [®]	Packaged	1	1.864	1.864	1.864	
9 Lives [®] Cat Food	Open	2	0.023	0.026	0.024	0.002
9 Lives [®] Cat Food	Packaged	1	0.027	0.027	0.027	
Whiskas [®] Cat Food	Open	2	0.100	0.109	0.105	0.006
Whiskas [®] Cat Food	Packaged	1	1.175	1.175	1.175	
Coffee Beans	Open	2	0.011	0.011	0.011	0.000
Ground Coffee	Open	2	0.799	0.832	0.816	0.023
Beef Jerky	Open	2	< 0.004	0.007	0.005	0.004
Beef Jerky	Packaged	2	< 0.004	< 0.004	< 0.004	0.000

31



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.4.1. Summary of Sulfuryl Fluoride Residue Data from Foods Fumigated with Sulfuryl Fluoride.

Commodity	Configuration	n	Sulfuryl Fluoride Residue Level, ppm [*]			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Powdered Nonfat Milk ²	Open	2	< 0.004	< 0.004	< 0.004	0.000
Powdered Whole Milk	Open	2	1.439	1.466	1.453	0.019
Powdered Cheese	Open	2	0.344	0.472	0.408	0.091
Powdered Eggs	Open	2	0.253	0.634	0.444	0.269
Garlic Powder	Open	2	< 0.004	< 0.004	< 0.004	0.000
Onion Powder	Open	2	< 0.004	< 0.004	< 0.004	0.000
Peppercorns	Open	2	0.012	0.018	0.015	0.004
Parsley	Open	2	0.120	0.204	0.162	0.059
Baking Powder	Open	2	0.027	0.036	0.032	0.007
Baking Soda	Open	2	< 0.004	< 0.004	< 0.004	0.000
Salt	Open	2	< 0.004	< 0.004	< 0.004	0.000
Sugar	Open	2	< 0.004	< 0.004	< 0.004	0.000
Basil	Open	2	0.008	0.013	0.010	0.004
Peanuts	Open	2	0.082	0.209	0.145	0.090
Chilis	Open	2	0.229	0.264	0.247	0.025
Ham	Open	2	< 0.004	< 0.004	< 0.004	0.000
Cocoa Beans	Open	2	0.113	0.121	0.117	0.006

^{*} Residue levels are corrected for concurrent recovery values.^a The Highest Average Field Trial (HAFT) for each table entry is equal to the maximum residue value due to the experimental design.^b The median (50th percentile) is not provided since there were, at most, only two residue values reported.

Shaded entries in the table are for commodity/analyte combinations with unacceptable method performance based on concurrent recovery.

Table C.4.2. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.

Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm [*]			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Cheezits®	Open	2	1.00	1.00	1.00	0.00
Cheezits®	Packaged	2	1.00	1.00	1.00	0.00
Fritos®	Open	2	4.42	5.61	5.02	0.84
Fritos®	Packaged	2	1.23	1.23	1.23	0.00
Doritos®	Open	2	5.94	5.95	5.95	0.01
Doritos®	Packaged	2	2.28	2.37	2.33	0.06
Spaghetti	Open	2	< 1	< 1	< 1	0.00
Spaghetti	Packaged	2	< 1	< 1	< 1	0.00
Egg Noodles	Open	2	17.50	17.80	17.65	0.21

32



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.4.2. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.						
Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm ^a			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Egg Noodles	Packaged	2	< 1	< 1	< 1	0.00
Chocolate Cake Mix	Open	2	6.72	7.86	7.29	0.81
Chocolate Cake Mix	Packaged	2	1.49	2.63	2.06	0.81
White Cake Mix	Open	2	2.77	7.28	5.03	3.19
White Cake Mix	Packaged	2	2.77	3.94	3.36	0.83
Corn Flakes	Open	2	1.38	5.27	3.33	2.75
Corn Flakes	Packaged	2	< 1	< 1	< 1	0.00
Granola	Open	2	16.01	18.71	17.36	1.91
Granola	Packaged	2	1.01	2.37	1.69	0.96
Flour Tortilla	Open	2	25.80	35.10	30.45	6.58
Flour Tortilla	Packaged	2	9.71	10.10	9.91	0.28
Corn Tortilla	Open	2	26.25	31.25	28.75	3.54
Corn Tortilla	Packaged	2	11.30	14.95	13.13	2.58
Pecan Sandies [®]	Open	2	15.70	17.10	16.40	0.99
Pecan Sandies [®]	Packaged	2	1.18	1.66	1.42	0.34
Peanut Butter Cookies	Open	2	56.73	67.73	62.23	7.78
Peanut Butter Cookies	Packaged	2	1.23	42.93	22.08	29.49
Coconut Flakes	Open	2	34.88	35.48	35.18	0.42
Coconut Flakes	Packaged	2	8.37	9.30	8.84	0.66
Oreo [®] Cookies	Open	2	25.60	27.00	26.30	0.99
Oreo [®] Cookies	Packaged	2	< 1	< 1	< 1	0.00
Alpo [®] Dog Food	Open	2	3.08	3.10	3.09	0.01
Alpo [®] Dog Food	Packaged	1	2.73	2.73	2.73	
Purina Puppy Chow [®]	Open	2	12.40	16.00	14.20	2.55
Purina Puppy Chow [®]	Packaged	1	19.20	19.20	19.20	
9 Lives [®] Cat Food	Open	2	8.87	9.93	9.40	0.75
9 Lives [®] Cat Food	Packaged	1	8.32	8.32	8.32	
Whiska's [®] Cat Food	Open	2	6.71	7.78	7.25	0.76
Whiska's [®] Cat Food	Packaged	1	4.93	4.93	4.93	
Coffee Beans	Open	2	6.81	7.48	7.15	0.47
Ground Coffee	Open	2	10.00	11.58	10.79	1.12
Beef Jerky	Open	2	24.72	37.92	31.32	9.33
Beef Jerky	Packaged	2	1.12	1.12	1.12	0.00
Powdered Nonfat Milk ²	Open	2	2.36	2.36	2.36	0.00

33



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

Table C.4.2. Summary of Fluoride Residue Data from Food fumigated with Sulfuryl Fluoride.

Commodity	Configuration	n	Fluoride Anion Residue Levels, ppm ^a			
			Min, ppm	Max, ppm ^a	Mean, ppm ^b	SD, ppm
Powdered Whole Milk	Open	2	1.07	2.31	1.69	0.88
Powdered Cheese	Open	2	4.05	4.90	4.48	0.60
Powdered Eggs	Open	2	690.84	819.84	755.34	91.22
Garlic Powder	Open	2	15.80	15.80	15.80	0.00
Onion Powder	Open	2	1.17	1.17	1.17	0.00
Peppercorns	Open	2	5.36	6.55	5.96	0.84
Parsley	Open	2	48.15	56.25	52.20	5.73
Baking Powder	Open	0				
Baking Soda	Open	2	14.10	15.60	14.85	1.06
Salt	Open	2	2.04	2.30	2.17	0.18
Sugar	Open	2	< 1	1.03	0.77	0.37
Basil	Open	2	48.83	53.53	51.18	3.32
Peanuts	Open	2	10.30	12.30	11.30	1.41
Chilis	Open	2	24.95	27.75	26.35	1.98
Ham	Open	2	17.43	18.73	18.08	0.92
Cocoa Beans	Open	2	12.30	14.01	13.16	1.21

^a Residue levels are corrected for concurrent recovery values.^a The Highest Average Field Trial (HAFT) for each table entry is equal to the maximum residue value due to the experimental design.^b The median (50th percentile) is not provided since there were, at most, only two residue values reported.

Shaded entries in the table are for commodity/analyte combinations with unacceptable method performance based on concurrent recovery.

D. CONCLUSION

Residue levels of sulfuryl fluoride and fluoride that result in foods following fumigation with sulfuryl fluoride are highly dependent on the properties of those foods, primarily fat and protein content. Furthermore, the performance of the analytical methods used in this study for sulfuryl fluoride and fluoride is not consistent across all of the foods tested and may be a factor in the observed large variability in the residue data. Fluoride may have become bound with food components during the interval that samples were stored, making it unavailable for analysis. Data are inconclusive regarding the ability of polymer film packaging materials to serve as a barrier to sulfuryl fluoride gas.

E. REFERENCES

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34



Sulfuryl fluoride/078003

DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3

Crop Field Trial - "Key" Ingredients, Finished Foods, and Pet Food

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F. DOCUMENT TRACKING

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40